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(54) **Isostatic pressing of sintered crushed spherical particles**

(57) A method for the production of shaped articles, particularly articles having irregular cross section, by hot isostatic compaction of metal powder, using as starting material a powder having substantially spherical grain shapes, crushing the substantially spherical powder to minor particles having irregular shape, forming a green body of the crushed powder, sintering the green body to obtain closed, that is to say non-communicating, pores, and subjecting the sintered body to hot isostatic compaction to full density. In an example, spherical particles of a high speed steel are wet milled under dichloromethane, dried, soft annealed at 780°C in vacuum lightly ground to disperse lumps, then cold compacted in a rubber mould at 4000 bar. The green body is then sintered at 1190°C and finally isostatically compacted at 1000 bar and 1150°C.

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SPECIFICATION

Method for the producti n of shaped obj cts

The present inv ntion relates to a method f r th production of shaped objects, particularly objects having irregular cross section, comprising isostatic hot compaction of metal powder.

If very small grains are made directly from molten metal, e.g. molten steel, each grain can be looked upon as a tiny ingot. Because the droplets which form the individual grains are cooled extremely rapidly, segregations in the grains are avoided such that the grains will have a very homogeneous composition. These conditions are well known and are utilized for the production of high grade steels susceptible to segregations, for example high speed steels. According to one method, known by the trade name ASP-method, steel powder of this type is produced by fragmentation of a jet of molten metal in inert gas atmosphere. The powder in the form of tiny spheres which have been protected against oxidation by the inert atmosphere during the production but also through its spherical shape which gives a low area/volume ratio, is poured into a thin walled plate capsule. The capsule is thereafter closed and the air is evacuated, whereafter the capsule and its contents are heated and subjected to isostatic compaction to full density. The billet made in this way is forged and thereafter rolled in conventional manner. The method brings about a starting material for the production of highly qualified tools with very good features and has therefore achieved increasing use.

It has also been proposed to produce, in a powder metallurgical way, tools with the configuration of the ultimate shaped tools to be produced. The advantage of this method as compared to the above briefly described method, which requires conventional working – plastic working as well as machining – is that several working steps can be eliminated, which may enable production at reduced costs. According to a proposed trend of development there is manufactured a sintered powder body into which an infiltration material is caused to infiltrate. This technique, however, can only be used for products with moderate demands as far as hardness and resistance to wear is concerned. For high speed steel tools for example, a homogeneous high speed steel body is looked upon as a prerequisite. In these cases therefore, the manufacturing requires hot isostatic compaction. According to one such method, which has to some extent been successful, a powder is produced by water atomization. The powder thereafter is heated to annealing temperature in order to remove oxides and also in order to reduce the hardness. Thereafter the powder can be cold pressed to the desired shape and be sintered to almost full density at such a high temperature that the structure of the material is influenced. This process may b suitable for the production f parts subjected to wear and other rather simpl tools but not for high speed steel to ls and th like, upon which are higher demands as far as strength and resistance to wear are made.

In the production of highly qualified products, such as high speed st l tools, to the ultimat shap from a metal powder ther seems to be a prerequisite that the starting mat rial is a powder with very low oxygen cont nt. A powder which can satisfy this d mand is that p wder with spherical shape which is used in the ASP-process. A problem in this context, however, is that this spherical powder cannot be pressed together to form a green body through cold isostatic pressing. It has therefore been proposed to arrange the powder in capsules of various kinds, e.g. in tailored plate containers, glass containers and the like. All these methods, however, involve steps which increase the total cost, and this is perhaps the major reason why the powder metallurgical production of highly qualified tools to ultimate shape has not made a break-through.

The present invention provides a method for the production of shaped objects, particularly objects having irregular cross section, by hot isostatic compaction of metal powder, which process comprises:

a) using as starting material a powder, the grains of which have substantially spherical shape,

b) crushing the powder to minor particles with irregular shape,

c) forming a green body from the crushed powder,

d) sintering the green body to produce closed, (non-communicating) pores, and

e) subjecting the sintered body to hot isostatic compaction to full density.

This method provides a means for powder metallurgical production of highly qualified tools and other articles to their ultimate shape or to a shape which at least approximately corresponds to the desired ultimate shape of the article.

According to the invention the starting material is a powder having substantially spherical shape of the individual grains. Suitably the powder consists of a gas atomized powder with low oxygen content, although in principle water atomized powder can also be considered, provided the powder is deoxidized prior to the continued treatment. For example one may advantageously utilize the method which is used in the ASP-process. According to this method nitrogen or argon is used as an atomization gas. The same gas also is contained in the cooling tower where the droplets solidify, whereby the melt, the droplets which are formed and the solidified powder are prevented from oxidizing. The method is disclosed e.g. in U.S. patent No. 3 813 196. In this manner the starting material for the present invention is advantageously obtained, said powder consisting of spherical particles with an average diameter between 100 and 200 μm. The oxygen content is preferably below 100ppm and normally of the order of 50 ppm.

The sperical powder which thus is produced is crushed according to the invention into minor particles with irregular shape. The crushing operati n may be disintegrated through the "cold stream method" which is known *per se*. According to this method the powder is cooled by liquid nitrogen such that it is extremely brittle. By means of the cooled gas the powder is blasted against a cooled wall such that it is crushed by impact. A disadvantage with this

technique, however, is that it is very energy consuming. Pref rably therefore the powder is disintegrated by wet milling without th access of air. As a milling liquid th re may b used dichloromethan (CH_2Cl_2), ethanol (C_2H_5OH) or oth r n n-oxidizing liquid with high vapour pressure, so that it may readily be removed after milling. Dichloromethane has its boiling point at 41°C.

Depending on the crushing method, the disintegrated powder may be dried in order to remove the milling liquid. The drying operation may cause the powder to be agglomerated into rather hard lumps. Therefore the dried material is desirably milled for a short while in the dry state in order to break down the lumps and reestablish the powder in the same state as before drying. The same type of mill may be used as in the wet milling operation.

After crushing, optional drying and after-treatment the powder is preferably soft annealed. The purpose of the soft annealing is to make the particles plastically deformable such that a green body of sufficient strength may be obtained through the following cold pressing operation. The soft annealing is performed without the presence of oxidizing gas, i.e. in a reducing atmosphere, in an atmosphere which is chemically inert as far as the metal powder is concerned, e.g. in nitrogen, or preferably, in vacuum. In order to achieve a very considerable reduction of hardness one may heat the powder to austenization temperature, which means about 850°C for high speed steel, whereupon the temperature is lowered past the perlite nose to below the Ac_1-temperature where the material is maintained for 10-20 h. A disadvantage with this method of soft annealing the powder is, however, that the very fine grain powder is readily agglomerated to very hard aggregates at the higher temperature. Therefore it is proposed, in spite of the fact that it will not give as high, but for the purpose however sufficient, hardness reduction, that the austenization treatment is eliminated. Instead the powder is heated from ambient temperature directly to a temperature immediately below its Ac_1-temperature such that the powder is soft annealed in the ferritic state so that there is obtained a metal powder which in the case of high speed steel has a structure substantially consisting of over-tempered martensite. A suitable holding time is 10-20 h.

The soft annealed powder thereafter may be charged in a compressible mould which has been shaped after a model. Principally a great number of various mould materials may be considered, such as metal, plastic etc. Preferably, however, the mould consists of rubber. Such a mould may readily be produced according to the art known *per se* by shell casting of rubber on a model, whereafter the mould is turned off. Prior to charging the powder into the mould one may supply a lubricant or binder, for example zinc stearate. Disadvantage with an agent such as zinc stearat however, is that the carbon in the steel powder may reduce the zinc which thereby is integrated into th powder alloy. As an alt rnative one may th refore also consider organic binders, which may remain in the powder until th sint ring temperature where they vaporize. Experiments, however, have nvisaged that by the combination of the crushing of the powder and th soft annealing th re is achi ved such a good self binding ability of the powder that th addition of additional bind rs prior to coldpressing compl tely may be avoided. The preferred mbodiment of the invention th refore is characterized in that the powder is cold pressed without the addition of additional binder.

Prior to cold pressing, the gas is evacuated from the mould which thereafter is sealed. The cold pressing therefore is performed in a manner which may be known *per se* by cold pressing in a water emulsion at a pressure exceeding 1000 bar, suitably exceeding 4000 bar. More particularly the cold pressing is performed as cold isostatic pressing which means that the pressing is performed at a temperature which is below the sintering temperature of the metal powder. Normally the cold pressing is performed at room temperature, but it is principally possible to carry out the pressing at higher temperature provided it is below the sintering temperature. This may in certain cases be advantageous. Thus it may render it possible to use plastic moulds which are stiff and have bad flexibility at room temperature but which at the higher temperature are more flexible and hence more compressible.

When the cold isostatic compaction is finished the mould is removed from the green body. In the case when the mould consists of rubber, the mould is turned off the green body, whereafter the mould can be repeatedly used. The stripped green body thereafter is sintered. If the green body contains a lubricant or binder this is first removed. The sintering is performed without any access of oxidizing gas. Preferably the sintering is performed in vacuum. The temperature and the sintering time is adapted to the composition of the present alloy, the grain size of the powder and the dimensions of the green body, so that there is obtained a sintered body with closed, that is to say non-communicating pores without coarsening the structure of the material. Generally speaking the sintering temperature should be immediately below the solidus temperature of the material, that is to say for high speed steel at a temperature between 1 100 and 1 300°C. The holding time normally is between 1 and 24 h. An advantage of the invention is that the holding time may be kept comparatively short by the fact that sufficient sintering effect is achieved rapidly due to the very high number of contact surfaces per unit of volume. This in turn means that any significant carbide growth can be avoided.

Finally the sintered body is hot isostatic compacted to 100% density. This is carried out in a manner known per se at a very high pressure which in existing pressing devices is in the order of 1 100 bar using argon as a pressing medium. The temperature is normally 1 000-1 200°C and the holding time about 1h. Possibly the sintered body is preheated prior to the h t isostatic compaction.

Also compound materials may b produced by the utilisation of the principles of the invention, in which case the powder may be provided n a core of solid material. As an alternativ it is also possible to provide powd r of diff rent types adjacent to each other and, according to the invention, cause these to be

combined into an article. The different parts of this article thus may obtain different ch mical composition and different features.

EXAMPLE

As a starting material there is used a gas atomized high speed steel powder, the spherical particals of which have average grain sizes of about 150 µm, and an oxygen content about 50 ppm. For example the high speed steel may be grade UHB® ASP® 23 having the nominal composition 1.27 C 4.2 Cr 5.0 Mo 6.4 W 3.1 V, balance iron and impurities (W-No 3344; AISI M3:2).

The powder is wet milled with the dichloromethane, CH_2Cl_2, as a milling liquid for 100h in a mill a suitable type. During laboratory tests a small mill of the rod mill type was favourably used. The milled product is dried in vacuum for expelling the milling liquid. By this drying operation there is obtained an agglomerate in the form of lumps which are milled under a short period of time, about 5 minutes, in the same mill in order to reestablish the fine powder. The powder is soft annealed at 780°C, 20 h, in vacuum. This operation again may cause a certain extent of agglomeration. In order to disintegrate this agglomerate there is used a mild tearing method. In the laboratory tests which were performed the lumps of powder were torn essentially without any plastic deformation by treatment by hand in a mortar. The powder thereafter is charged in the rubber mould shaped after the present model. The gas in the mould is evacuated and the mould is thereafter sealed and the mould with its content of powder is cold compacted at 4 000 bar during 10 minutes under full pressure, totally 1 h. The rubber form thereafter is turned off and the green body is sintered in vacuum, 1190°C, 1 h. The sintered body is cooled to ambient temperature and preheated to 1150°C and is hot isostatic compacted at 1000 bar, 1150°C, 1 h. As a result there is achieved a body free from pores, big slag inclusions and big carbides.

CLAIMS

1. A method for the production of shaped objects, particularly objects having irregular cross section, by hot isostatic compaction of metal powder, which process comprises:

a) using as a starting material a powder, the grains of which have substantially spherical shape,

b) crushing the powder to minor particles with irregular shape,

c) forming a green body from the crushed powder,

d) sintering the green body to produce closed, (non-communicating) pores, and

e) subjecting the sintered body to hot isostatic compaction to full density.

2. A method according to claim 1 in which the starting material is a gas atomized powder with low oxygen c ntent.

3. A method according to claim 1 or 2, in which in b) th powd r is crushed ess ntially without adoption of oxygen.

4. A method according to any on of th preceding claims in which a lubricant is mixed with th crushed powder prior to forming the green body.

5. A method according to claim 4 in which the lubricant is zinc stearate.

6. A method according to any on of th preceding claims in which, prior t forming the green body, th powder is provided in a casing, th interi r of which has the desired shape of the article to be produced.

7. A method according to claim 6 in which the casing is a rubber mould.

8. A method according to any one of the preceding claims in which the crushed powder is pressed to form a green body.

9. A method according to claim 8 in which the green body is formed by cold isostatic pressing.

10. A method according to any one of the preceding claims in which the green body is sintered in at least two steps, wherein during an initial step any lubricant is expelled and during one or more subsequent steps a sintering effect is achieved to produce closed (non-communicating) pores without coarsening the structure of the material.

11. A method according to any one of preceding claims, in which the powder consists of a high speed steel powder.

12. A method according to any one of claims 9 to 11 in which the crushed powder is soft annealed prior to cold isostatic compaction.

13. A method according to claim 12 in which the cold isostatic compaction of the crushed and soft annealed powder is performed without any extra binder.

14. A method according to any one of the preceding claims in which the powder is provided on a core of solid material or in association with a different powder to form a compound material.

15. A method according to any one of the preceding claims in which the shaped object is a high speed steel tool.

16. A method according to claim 1 substantially as described in the foregoing Example.

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